28

HEAT-STERILIZABLE, REMOTELY ACTIVATED BATTERY DEVELOPMENT PROGRAM PHASE II Fourth Quarterly Report

Period: April 1, 1969 to June 30, 1969

Contract No. 952214 NAS7-100

July 25, 1969

JET PROPULSION LABORATORY California Institute of Technology 4800 Oak Grove Drive Pasadena, California '91103

ELECTRONICS DIVISION
Eagle-Picher Industries, Inc.
Couples Department
Joplin, Missouri



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"This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, as sponsored by the National Aeronautics and Space Administration under Contract NAS7-100."

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ABSTRACT

This report discusses the progress during the fourth quarter of the development of a heat-sterilizable, sealed, remotely activated battery per contract number 952214 between The Jet Propulsion Laboratory and Eagle-Picher Industries, Inc. The work is moving to the completion of Phase I and initiation of Phase II. Phase I is defined as an investigation of basic cell and battery components. Phase II consists of the assembly of the components selected from Phase I into a battery.

Studies pertaining to the silver-oxide electrode have shown that prior to sterilization the crystalline structure is irregular and has no similarity between plates. After sterilization the plates become more uniform on a plate-to-plate comparison but are less uniform across the surface of an individual plate. X-Ray analysis revealed that prior to sterilization the only phase present was AgO. The predominant phases found after sterilization were Ag and Ag₂O with a small amount of AgCO₂.

The zinc electrode was found to be physically stable through heat sterilization. This was verified through microscope scanning shots, X-Ray measurements and B.E.T. surface area measurements.

Due to heat sterilization the resistivity of the separator is reduced to a level slightly above that which would be expected after 24 hours immersion in 1.30 specific gravity potassium hydroxide.

Mechanical tests conducted with molded polysulfone cell cases indicate a 40% loss in strength due to $135\,^{\circ}\text{C}$ heat sterilization.

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1.0 INTRODUCTION

This report presents the results of work during the fourth quarter ending 30 June 1969. The individual components necessary for a remotely activated heat-sterilized battery were studied. Also, some preliminary design and testing of cells were accomplished.

2.0 TECHNICAL DISCUSSION

2.1 Silver-Oxide Electrode

During the past quarter studies were made to determine the effects of heat sterilization on various physical parameters of the silver-oxide electrode. These included crystalline structure, porosity, and chemical compounds. The methods used were microscope scanning, X-Ray diffraction, and B.E.T. measurements. Additional measurements were made of the weight losses and increases in thickness. These are shown in Table I.

The work pertaining to the crystalline structure and phase changes was performed by the University of Missouri at Rolla. The B.E.T. tests were performed by American Instruments Company. Analysis was made on plates before and after sterilization.

Microscopic examination was made of nine electrodes representing four weight groups. Each was compressed to a different density as is indicated by the thickness variations shown in Table I. The views included transverse as well as flat surface shots of the plates. Magnifications of 300% and 1000% were used. The pictures are shown in Appendix I. Evaluation of the scanning microscope shots was made without knowing the thickness so as not to influence the conclusions.

The transverse shots revealed irregularities or variations in density through the thickness of the electrodes. This was due to the pressing operation and pressure variations through the thickness caused by both depth and the presence of the silver grid. In general, however, the electrode structure was fairly uniform.

TABLE I
EFFECTS OF STERILIZATION
ON
SILVER-OXIDE AND ZINC
ELECTRODES

Plate		Weig		%		kness	%
No.	Туре	Before	After ————	Change'	Before	After	Change
35	Silver	6.604	6.314	-4.3	.026	.029	+11.5
106	11	6.698	6.387	-4.6	.028	.035	+25.0
83	11	6.594	6.286	-4.7	.028	.033	+17.9
95	IT	7.537	7.184	-4.7	.028	.033	+17.9
120	11	6.248	5.978	-4.3	.025	.029	+16.0
171	IT	6.131	5.848	-4.6	.031	.036	+16.1
206	11	8.258	7.890	~4.5	.033	.037	+12.1
2 52	11	8.452	8.101	-4.2	.028	.030	+ 7.1
78	Zinc	3.993 [°]	3.969	-0.6	.030	.030	+ 0.0
162	H	3.255	3.239	-0.5	.031	.032	+ 3.2

2.1 Silver-Oxide Electrode (Continued)

One noteworthy feature is the very angular nature and large overall size of the AgO at lower weights and density. The porosity or void area is also extensive for these electrodes. Both the structure and void area change considerably with increased weight and density. The angularity is not evident and the particles tend to form a more continuous mass, made up of smaller, more rounded agglomerated particles.

The angular particles ranged from a maximum in the range of 10 microns for one particle down to one micron or less. The smaller particles appeared to be more agglomerated, whereas the larger ones retained their identity and remained more separated from the mass. No two specimens were necessarily alike, but arrangement into a scale from very angular and open to agglomerated and dense would be possible.

In some cases it was difficult to obtain a clean, even break on the transverse sections, so it is possible that a few of the pictures could have been misleading.

The sterilized samples were very brittle and susceptible to crumbling. Because of the difficulty in getting a good sample, only surface shots were made.

The structures of all the samples appeared to become much more similar after sterilization. An overall agglomeration occurred with less apparent void space. There were no longer any of the individual angular particles that were present initially. All the particles appeared to be made up of a large number of small particles of about micron size.

The particles appear to result from some growth mechanism outward from the starting particle. All the electrodes after heating approached the structure that the denser, heavier

2.1 Silver-Oxide Electrode (Continued)

plates possessed before heating - namely, the agglomerated small particle-type structure. There was also a needle type growth in a few areas on some of the electrodes.

In general, there was much less uniformity over the entire surface area of a sterilized electrode than in the initial unsterilized plates, even though all the samples approached the same type. In other words, the majority of the electrodes will have similar areas, but the color, phases, and structure are more prone to vary within any one given electrode.

X-Rays were made primarily on one set of electrodes with common weights but varying densities. Patterns were made on the whole surface as well as of powders made by grinding up pieces taken from the electrodes.

Initially, the only phase present was AgO. After sterilization at 135°C for 128 hours, the samples appeared to be of a uniform dark to black color. Upon cooling a more mottled look appeared. The colors ranged from a light tan to intermediate shades of grey-brown to almost black. In examining surface X-Rays of sterilized plates, the samples were different in many respects. The powdered samples of these materials were very similar. This would indicate that either only the surface has any variations, or that the method of preparing the powdered specimens causes preferred phases to be collected. For example, a brittle phase (oxide, carbonate) would remain fine grained and powdery, while a ductile phase (silver) would agglomerate and flatten out being less likely to remain on the glass slide used to hold the powder for X-Ray.

The surface samples appear to be predominantly Ag and ${\rm Ag}_2{\rm O}$, with possibly a small amount of ${\rm AgCO}_3$. When powdered, a considerable amount of ${\rm AgCO}_3$ was found to be present. This may

2.1 Silver-Oxide Electrode (Continued)

not be representative or directly proportional to the amount in the plate, but it does show that this phase is present in all three. Evidently the carbonate was introduced during the sterilization of the plates.

The results of the B.E.T. tests conducted by American Instruments are not complete as yet. These involve measurements of the surface area of the electrodes. Effects of 128 hours of sterilization at 135°C on the surface area should be seen by these tests. Preliminary information indicates that the area is affected but the extent is not known. The data shall be presented when testing is completed.

2.2 Zinc Electrode

Coincidental with the studies made on the silver-oxide electrode, determinations of the effects of heat sterilization on the crystalline structure, porosity and surface area of the zinc electrode were made. Phase study determinations as well as microscope scanning shots (Appendix II) were made of several plates. There did not appear to be an appreciable oxidation of the zinc electrodes resulting from heating the plates at 135°C for 128 hours. The X-Rays taken before and after are almost identical, with only a Zn phase appearing. There was no evidence of any extensive amount of a ZnO phase. The weight changes were minor. However, the trend appeared to be inverse to that experienced before. All previous tests resulted in a weight increase and capacity loss. Since the two factors occurred coincidentally, the gain in weight was suspected as being oxygen. These data show essentially no significant weight changes during sterilization. Since the explanation for the lack of expected weight increase is not known, the tests are being repeated.

The tests performed at American Instruments indicate very little change, if any, in the surface area of the zinc plate after sterilization. The results of a two plate study are shown in

2.2 Zinc Electrode (Continued)

Table II. The variances between the unsterilized and sterilized readings are within the error of the technique.

2.3 Separator

Values for the resistance of several open type separators were determined during the past quarter. The separators were placed in the fixture shown in Figure II for measurement. The measurements were made with a 1000 Hz A.C. voltage and a fixed current of 0.5 ampere. Voltage readings were taken of the voltage drop across the electrolyte with no separator. The separator was then positioned in the fixture and voltage readings were taken again. Both measurements were recorded with a current of 0.5 amp.

The material used represented sterilized and unsterilized conditions. All of the materials previously tested were not readily available. However, the samples examined provide a representation of the various types of open separators. Two layers of the separator material were used in order to obtain a voltage differential sufficient to determine accurate values for the resistance. Since the materials used were essentially open separators, the value of resistance with respect to the number of layers should be relatively linear.

The results of the study are depicted in Table III. The values of resistance for unsterilized material ranged from 0.002 ohm to 0.029 ohm. Sterilized separator resistances had a spread of 0.002 ohm to 0.029 ohm. Frequently, the resistance calculated at 24 hours was higher for sterilized materials. Apparently, the 135°C sterilization made the separator more stable resulting in a relatively constant resistance.

2.4 Cell Case and Sealing

Various mechanical tests were conducted with polysulfone material. These included tensile and shear tests (lap joints) on fabricated components. Burst tests were conducted on molded cell cases. The specimens were stressed in unsterilized and sterilized conditions.

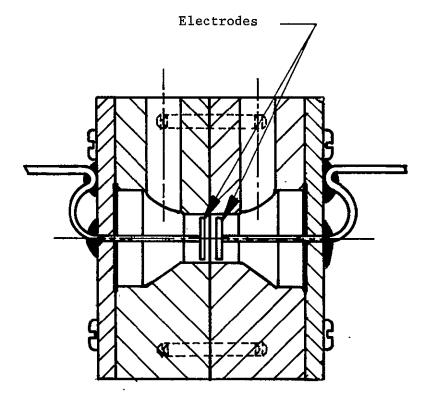


TABLE II

EFFECTS OF 135°C STERILIZATION

ON THE

SURFACE AREA OF ZINC PLATES

Plate No.	Condition	B.E.T. Measurement (Square Meters/Gram)
43	Unsterilized	6.1
	Sterilized	5.8
312	Unsterilized	3.6
	Sterilized	4.8

TABLE III EFFECTS OF HEAT STERILIZATION ON THE RESISTANCE OF SEPARATOR MATERIAL

Sample				Layers		ance/Laye		Status
No.	Туре	Supplier	E*	Tested			24 Hrs.	Status
1	Asbestos 7410 40% Glass	Raybestos	.13	2	0.0235	0.0235	0.0039	Unsterilized
2			.15	2	0.037	0.029	0.029	Sterilized
	Asbestos 7410/5 40% Glass	Raybestos	.03	2	0.0058	0.0058	0.002	Unsterilized
		 	.03	2	0.0058	0.0058	0.0039	Sterilized
	Asbestos 7401/5 5% Binder	Raybestos	.03	2	0.0058	0.0058	0.002	Unsterilized
<u>6</u> 7		 	.02	2	0.0058	0.0039	0.0039	Sterilized
7	Asbestos 7401 5% Binder	Raybestos	.02	2	0.0039	0.0039	0.002	Unsterilized
<u>8</u> 9		•	.01	2	0.002	0.002	0.002	Sterilized
	Nylon/B 2504K	Pellon Corp.	.03	2	0.0058	0.0058	0.0039	Unsterilized
10 11		Pellon Corp.	.02	2	0.0058	0.0039	0.0039	Sterilized
	Nylon/B 2505	Pellon Corp.	.04	2	0.0098	0.0078	0.0058	Unsterilized
12			.03	2	0.0058	0.0058	0.0058	Sterilized
	Nylon/B 2505K	Pellon Corp.		2	0.0078	0.0078	0.0058	Unsterilized
14 15		Pellon Corp.	.05	2	0.0118	0.0098	0.0098	Sterilized
	Nylon/B 2505ML	Pellon Corp.		2	0.0078	0.0078	0.0039	Unsterilized
16 17		,	.08	2 2	0.0156	0.0078	0.0078	Sterilized
	Ny1on/B 2506K	Pellon Corp.	.08	2	0.0156	0.0156	0.0118	Unsterilized-
18 19	······································	Pellon Corp.	.07	<u>2</u> 2	0.0137	0.0137	0.0137	Sterilized
	Nylon/B 2506M	Pellon Corp.	.04	2	0.037	0.0078	0.0058	Unsterilized
<u>20</u> 21			.11	2 2	0.035	0.0215	0.0196	Sterilized
21	Nylon/PP SM124.3	Kendall Mills	3.05	2	0.0118	0.0098	0.0098	Sterilized
22	Polypropylene EM476	Kendall Mills	.06	2	0.0137	0.0118	0.0098	Unsterilized
23	Polypropylene 2530	Pellon Corp.	.08	2	0.0196	0.0196	0.0137	Unsterilized
24	Rayon R75D	Chicopee Mills	.04	2	0.0078	0.0078	0.0058	Unsterilized
25			.04	2	0.0098	0.0078	0.0078	Sterilized
26	Cotton 2409	Kendall Mills	. 03	2	0.0058	0.0058	0.0039	Unsterilized
27			.06	2	0.0118	0.0058	0.0058	Sterilized
28	Hemp Filpaco 4366T	Dexter	.02	2	0.0039	0.0039	0.002	Unsterilized
29			.01	2	0.002	0.002	0.002	Sterilized

^{*}This is the differential voltage between readings taken without separator and with separator

2.4 <u>Cell Case and Sealing (Continued)</u>

Tensile and shear specimens were prepared using methylene chloride as the bonding agent. In the direct tensile samples the material was tapered to provide a joint approximately 0.5 inch in width. The material thickness was 0.125 inch. The shear specimens were made with an overlap of approximately 0.6 inch. Each sample was 1.0 inch in width. The pieces were broken using conventional test equipment and a steady crosshead movement. The parts were positioned in the jaws according to the type information desired. The manner in which the shear tests were held introduced some bending in the part. These samples all broke in an area adjacent to the lap joint. The load at the time of fracture ranged between 320 to 550 pounds. A rough calculation assuming strictly tensile loading indicates a breaking strength of 2500 to 4300 psi. The sterilized specimens were in the mid-range of the data as depicted in Table IV. The samples tested in tension broke within a range of 100 to 300 pounds. This represents stresses of 1660 to 5330 psi. The data from the sterilized parts were in the upper half of the range. Apparently, the joints were not made with sufficient pressure since the strengths of the tensile specimens are low.

A more definitive test of the polysulfone material was made using molded cell cases. Four cases were ruptured with nitrogen. Two of the four were sterilized prior to rupture. Each case had been sealed with methylene chloride and subjected to 85°C vacuum for 72 hours prior to testing. While the parts were examined visually for any crazing, a steady pressure increase was used to break the case. In each case the part failed without any crazing being seen. The major area exposed to pressure was 1.37 inches wide by 2.828 inches high with a wall thickness of 0.068 inch. The cases which had been sterilized failed at 85 and 90 psig while the unsterilized broke

TABLE IV

MECHANICAL STRENGTHS OF STERILIZED AND UNSTERILIZED POLYSULFONE JOINTS

Sample	Area of Fracture (Sq. In.)	Load at Failure (psi)	Stress (psi)	Sterilized	Remarks
1	.128	430	3360	Yes	Lap joint, failed adjacent to joint
2	.127	460	3630	No	Lap joint, failed adjacent to joint
3	.128	320	2500	Yes	Lap joint, failed adjacent to joint
4	.128	550	4300	No	Lap joint, failed adjacent to joint.
5	.0631	200 .	3170	Yes	Butt joint, failed at joint
6	.0562	300	5330	Yes	Butt joint, failed at joint
7	.0602	100	1660	No	Butt joint, failed at joint
. 8	.0610	200	3280	No	Butt joint, failed at joint
9	2.828 x 1.375	85	*	Yes	Cell case, failed at wall described
10	2.828 x 1.375	90	*	Yes	Cell case, failed at wall described
11	2.828 x 1.375	175	*	No	Cell case, failed at wall described
12	2.828 x 1.375	140	*	No	Cell case, failed at wall described

* - Value Not Determined

2.4 Cell Case and Sealing (Continued)

at 140 and 175 psig. Apparently, sterilization effected a loss in strength of approximately 40%. Each part failed in the manner expected.

2.5 Pyrotechnics

As a result of a continuation of studies involving pyrotechnics, an initiator developed by Networks Electronics utilizing pyrofuse as the bridgewire has shown evidence of being capable of heat sterilization. Five of these units were subjected to 135°C heat sterilization for 128 hours. After sterilization a no-fire current of 0.5 ampere was imposed on each igniter for 16 seconds followed by a sure-fire current of 2.0 amperes. No malfunctions occurred.

2.6 Cell Design

Entering into Phase II of the program, various cell designs were prepared for testing. The capacity requirements are either a 1200 watt rate for 10 minutes or a 500 watt rate for 24 minutes. The terminal voltage of the battery should be in the range of 26 to 30 volts when under the referenced loads. One design was selected for testing to verify the capacity.

Five cells were made using five negative and four positive plates separated by asbestos (.010 inch thickness) and 4366T hemp fiber (.0045 inch thickness). The cells were encased in fabricated polysulfone cases and sterilized 128 hours at 135°C while dry. Activated with 1.30 specific gravity potassium hydroxide, the cells were discharged according to the following schedule.

Cell No.	Wet Stand (Hours)	Discharge Rate (Amperes)
1	0.5	19.2
2	0.5	46.2
3	72	46.2 - 5 Min. & 19.2
4	144	46.2 - 5 Min. & 19.2
5	288	46.2 - 5 Min. & 19.2

2.6 Cell Design (Continued)

In each case the cells were marginal in capacity. Examination of the data (Figures II through V) and the expended cells revealed a shortage of negative material in the cell. The fifth cell failed to respond when the discharge load was applied. Evidently silver penetration of the separator had decreased the resistance of the separator sufficiently to permit the cell to discharge during the wet stand.

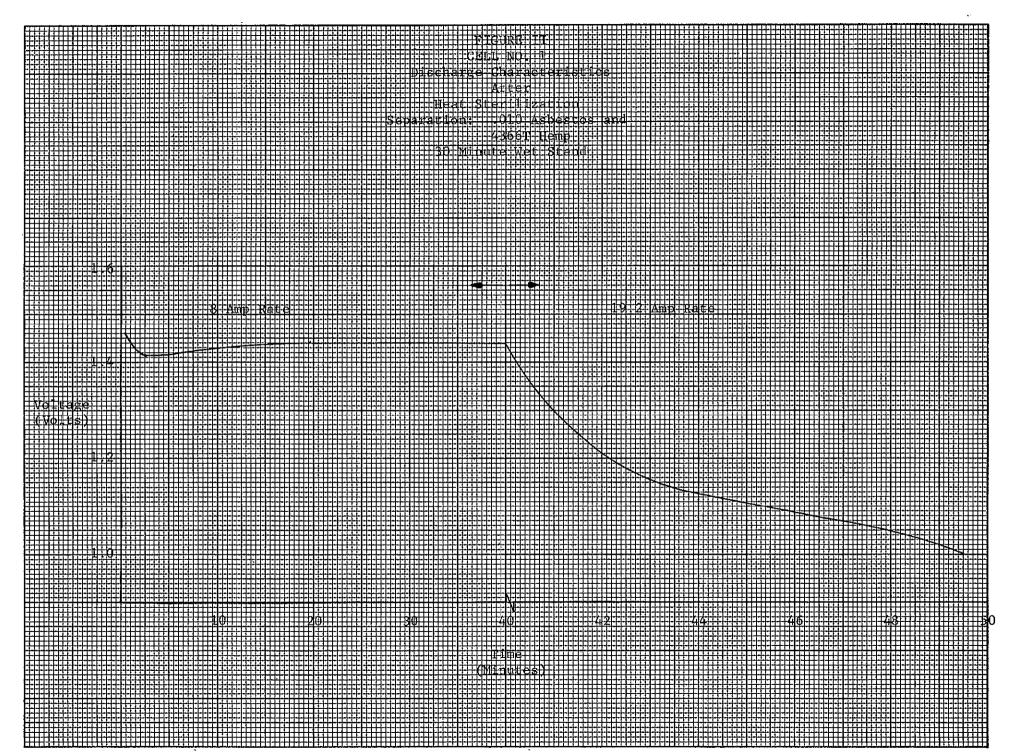
Two cells were made and discharged with each of the three inside negative plates replaced by two plates. Each of the two plates were the same as the one replaced. The cells were activated and allowed to stand for 0.5 hour prior to discharge. One cell was discharged at 46.2 amps while the other was discharged at 19.2 amps. Curves depicting the voltage-time profiles may be seen in Figures VI and VII. As can be seen by the curves, the marginal capacity condition has been eliminated by the increase in negative material.

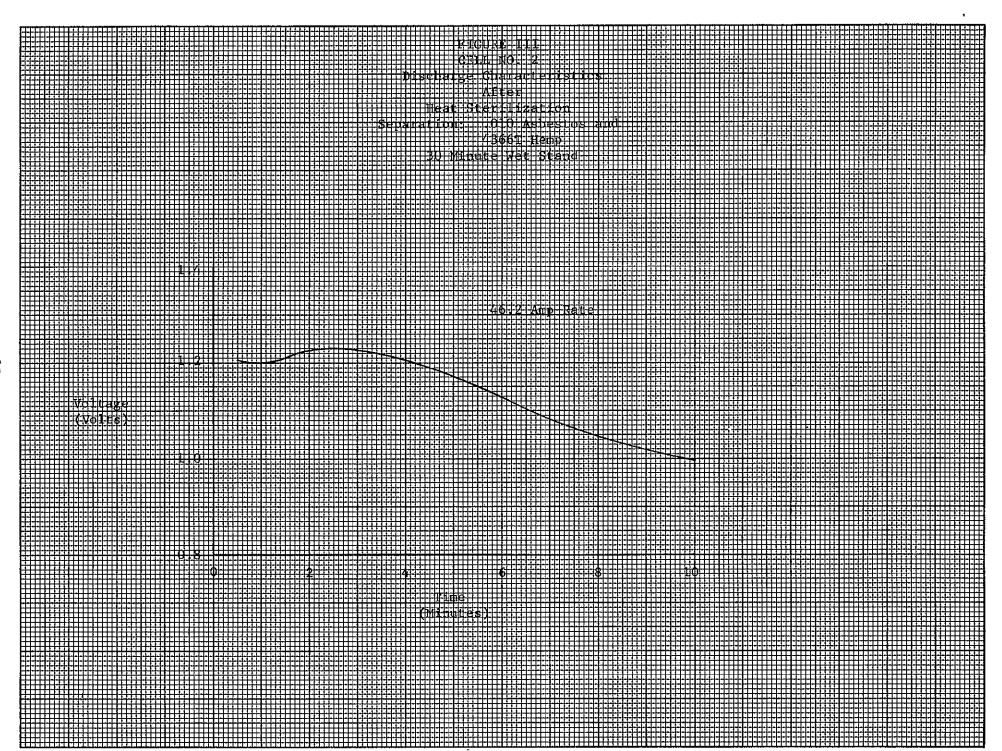
3.0 CONCLUSIONS

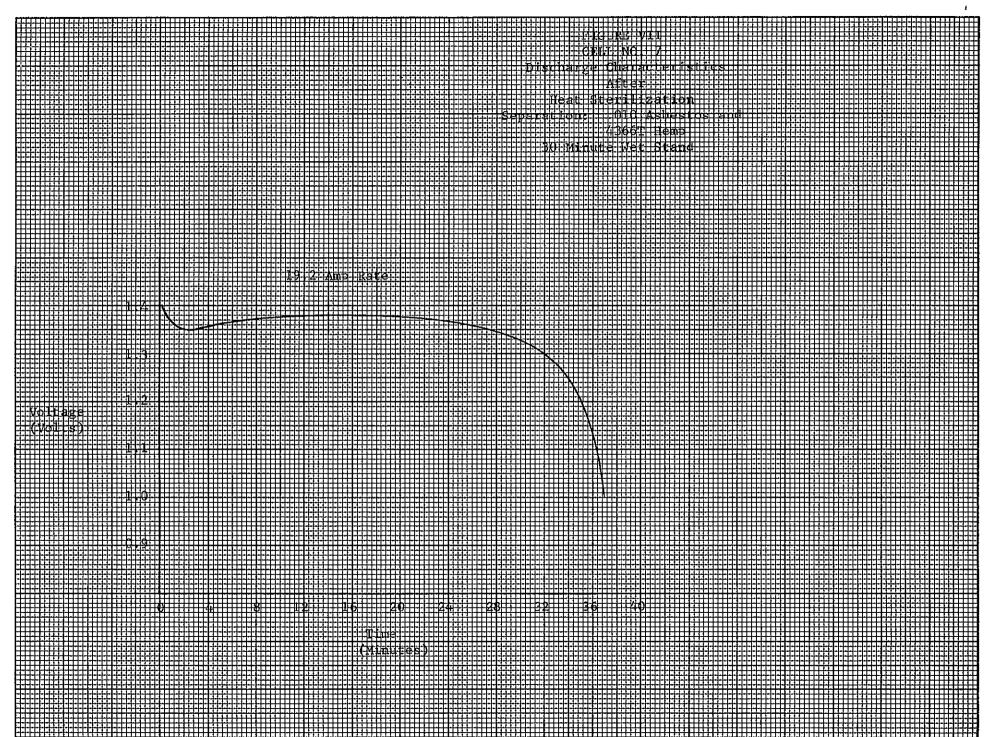
As a result of studies made during the past quarter, the following conclusions may be made. Prior to sterilization, the crystalline structure of the silver-oxide electrode is irregular. On a plate-to-plate comparison there is no similarity. After sterilization the electrodes become more uniform when compared on a plate-to-plate basis. However, the surface areas of individual plates were found to be less uniform after sterilization.

Prior to heat sterilization, the only phase present on the silver-oxide plates was determined to be AgO. Subsequent to sterilization the predominant phases found by X-Ray were Ag and ${\rm Ag}_2{\rm O}$, with a small amount of ${\rm AgCO}_3$.

Microscope scanning of zinc electrodes indicates that no significant changes occur in the physical structure as a result of heat sterilization. This was verified by B.E.T. surface area measurements.







3.0 CONCLUSIONS (Continued)

The resistances of the unsterilized separator materials measured as a function of time immersed in potassium hydroxide (1.30 specific gravity) were almost always higher initially than at the 24 hour point. After sterilization the resistance tends to stabilize at the lower levels.

Burst tests conducted on molded polysulfone cell cases indicate a 40% loss in strength due to sterilization.

4.0 RECOMMENDATIONS

Work will continue under Phase II.

5.0 NEW TECHNOLOGY

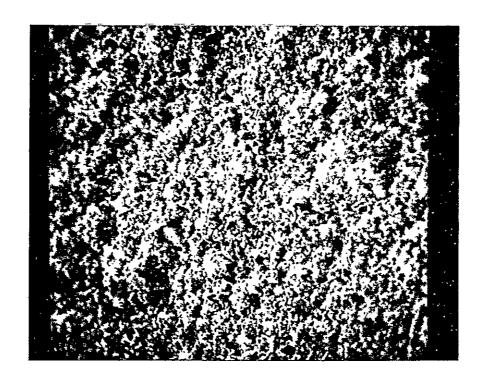
No new technology was developed during the fourth quarter of the contract.

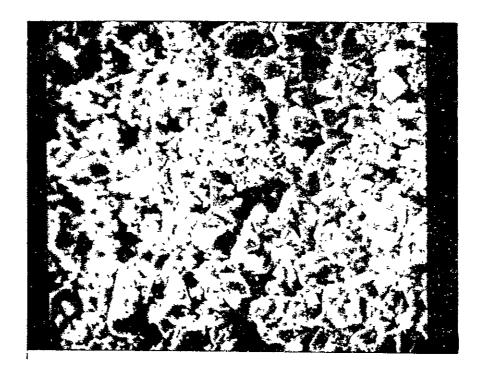
APPENDIX I . MICROSCOPIC STUDY OF SILVER-OXIDE ELECTRODES

Silver Oxide Plate No. 35

As Received

Flat Surface

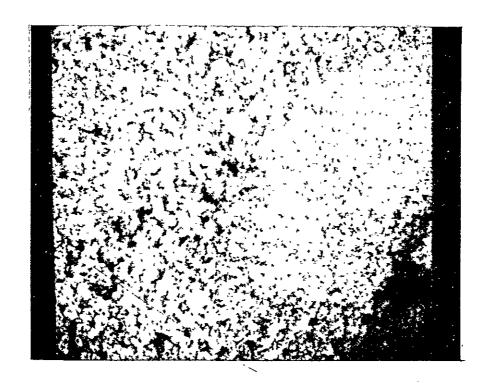


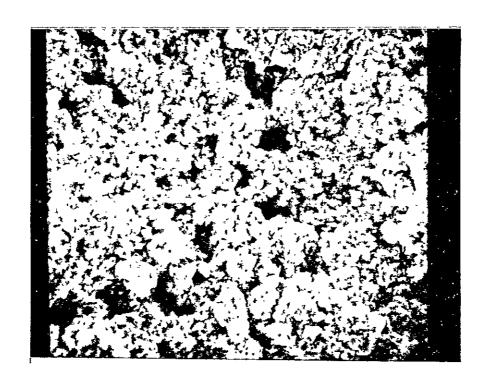


Silver Oxide Plate No. 35

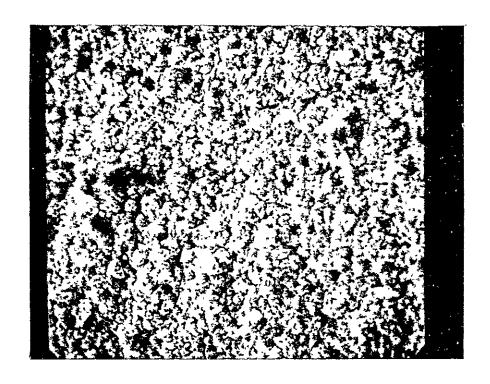
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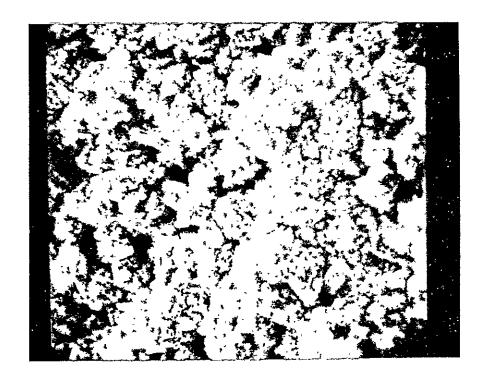
Transverse View





Silver Oxide Plate No. 35
Sterilized
Flat Surface

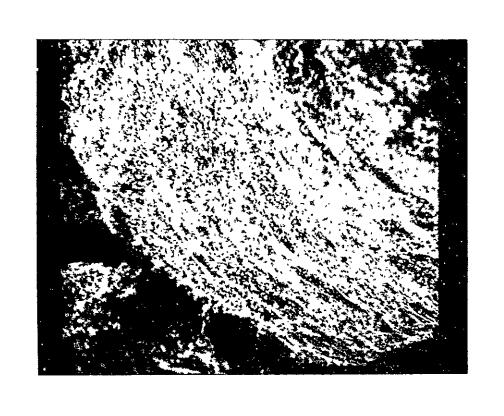


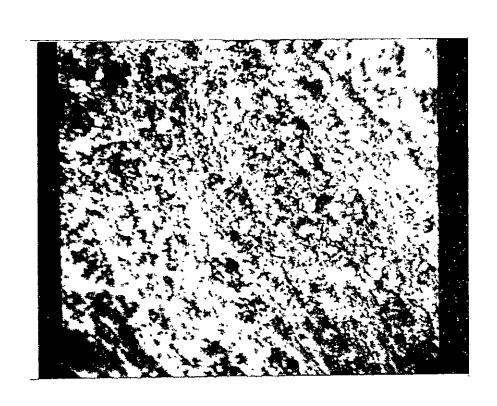


Silver Oxide Plate No. 67

As Received

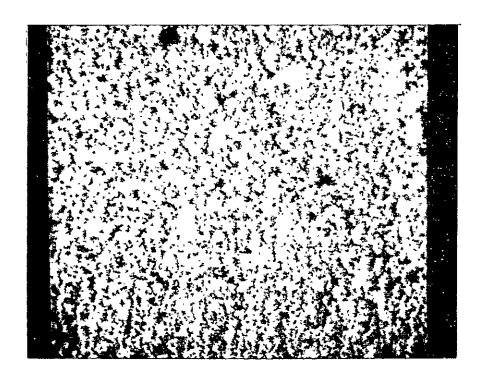
Transverse View

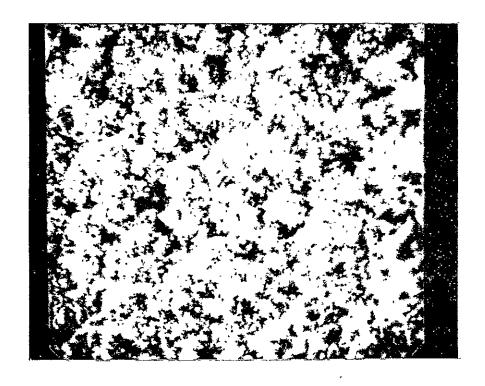




NOT REPRODUCIBLE

Silver Oxide Plate No. 67
Sterilized
Flat Surface

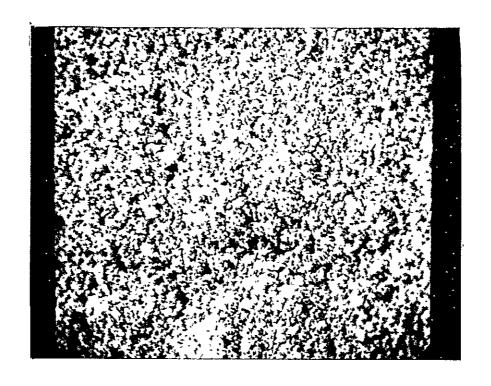


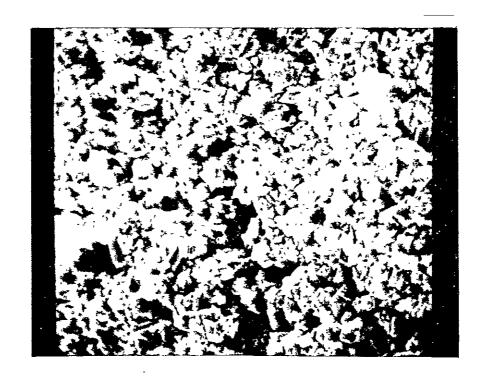


Silver Oxide Plate No. 83

As Received

Flat Surface

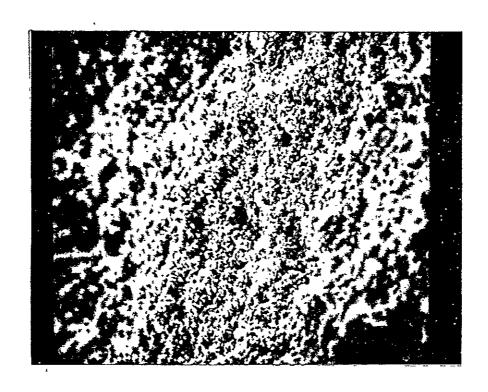


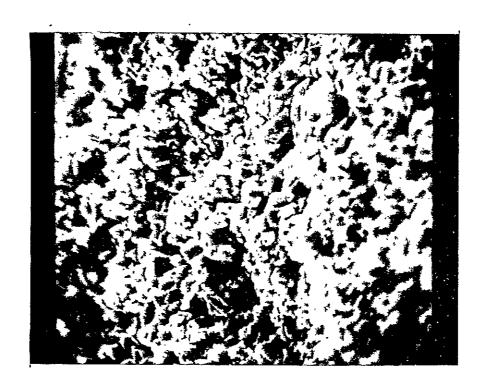


Silver Oxide Plate No. 83

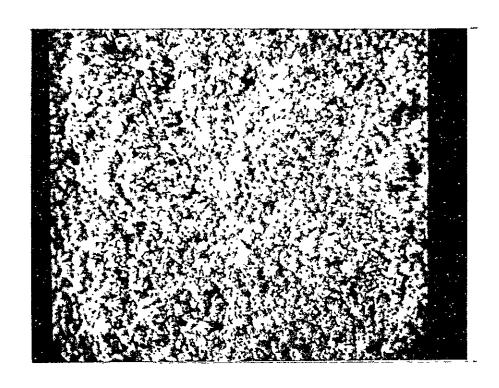
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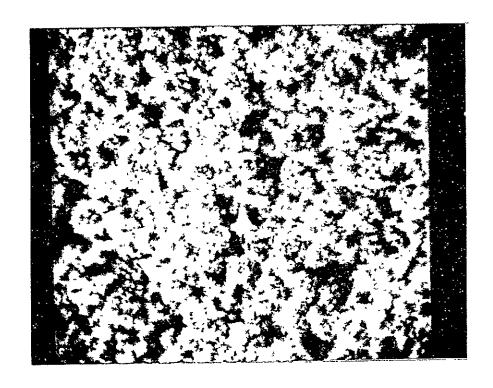
Transverse View





Silver Oxide Plate No. 83
Sterilized
Flat Surface

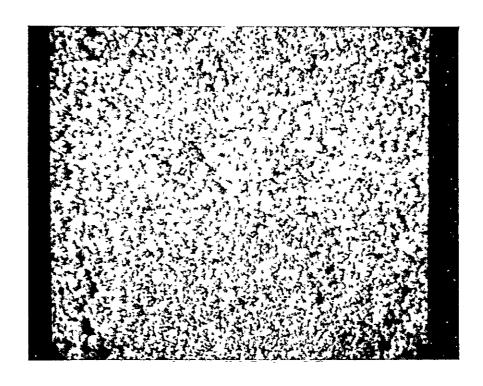


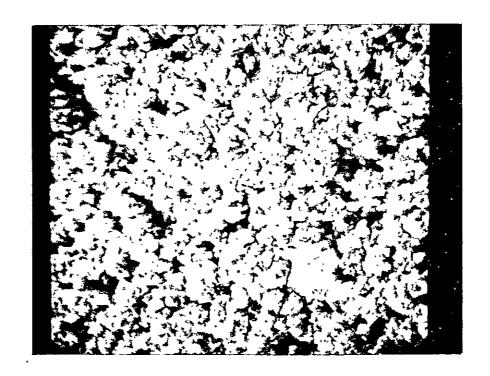


Silver Oxide Plate No. 95

As Received

Flat Surface

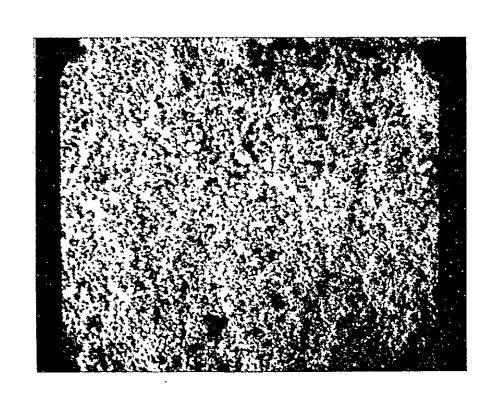


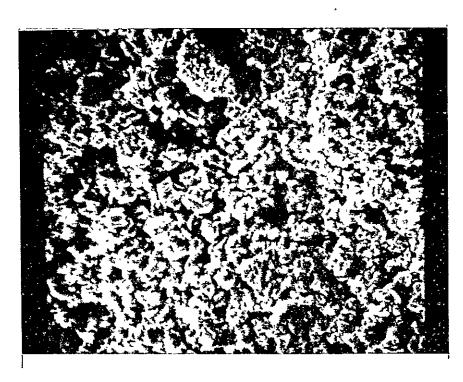


Silver Oxide Plate No. 95

As Received

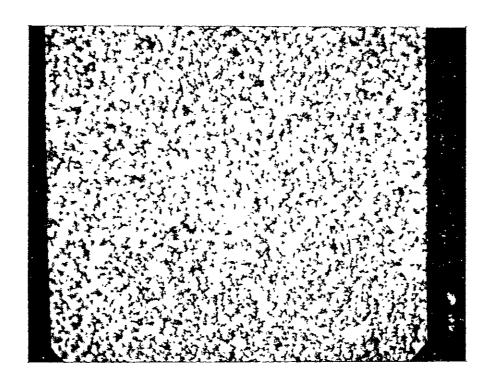
Transverse View

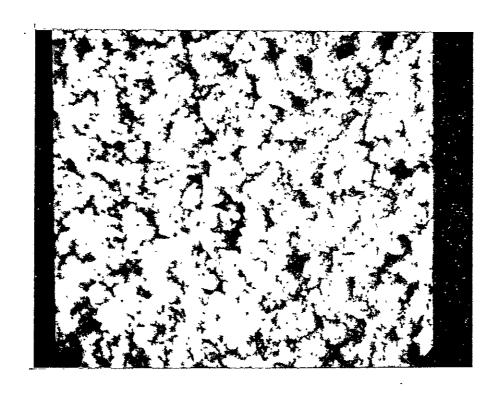




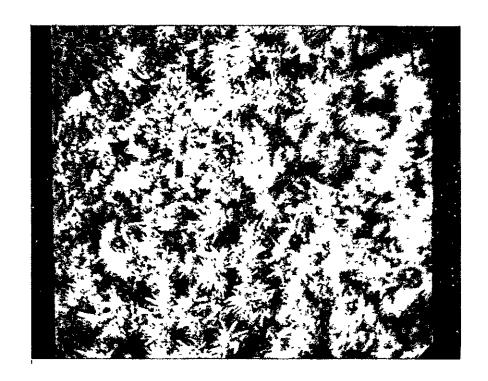
NOT REPRODUCIBLE

Silver Oxide Plate No. 95
Sterilized
Flat Surface





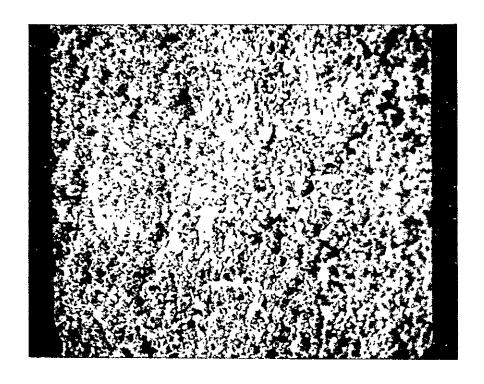
1000 Magnification
Silver Oxide Plate No. 95
Sterilized
Flat Surface

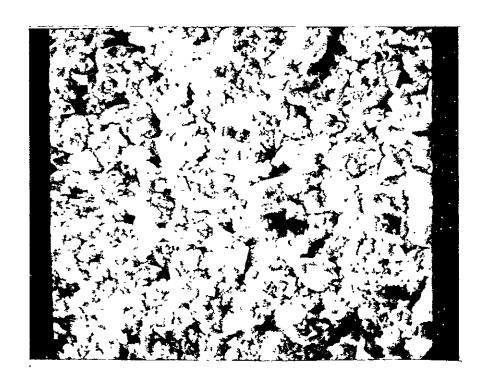


Silver Oxide Plate No. 106

As Received

Flat Surface

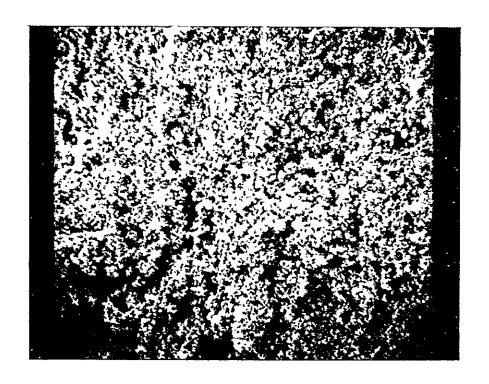


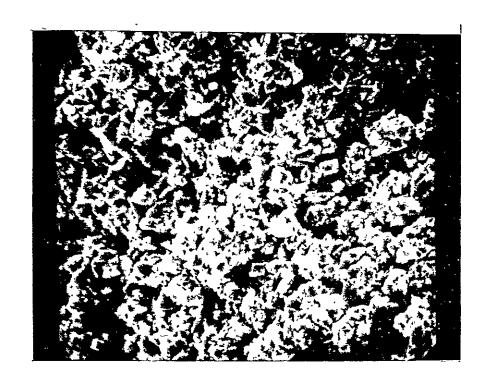


Silver Oxide Plate No. 106

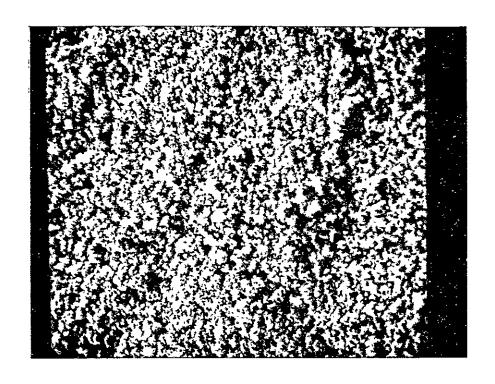
As Received

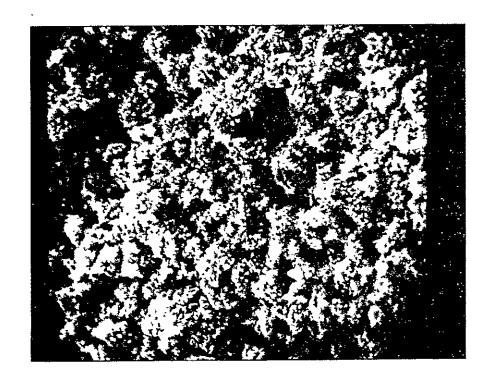
Transverse View





Silver Oxide Plate No. 106
Sterilized
Flat Surface

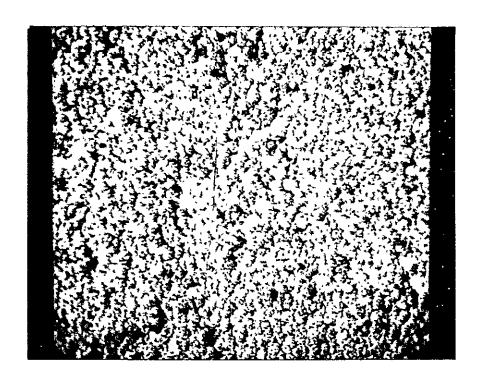


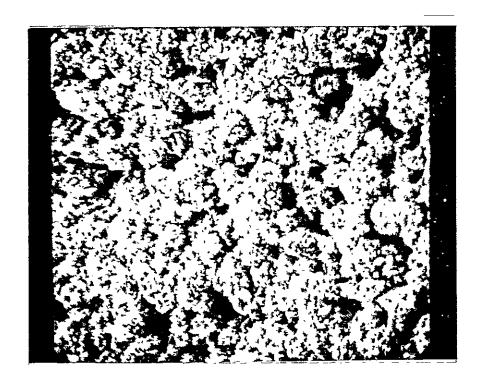


Silver Oxide Plate No. 120

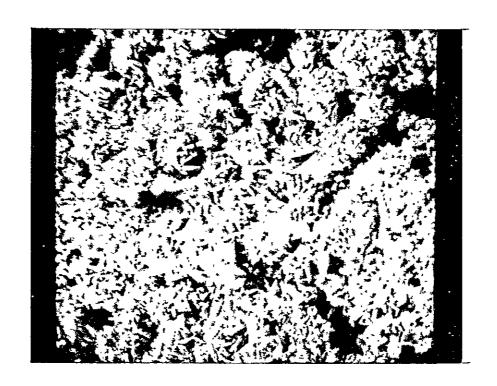
As Received

Flat Surface





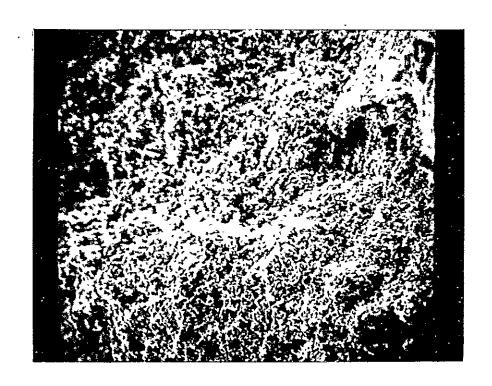
1000 Magnification
Silver Oxide Plate No. 120
As Received
Flat Surface

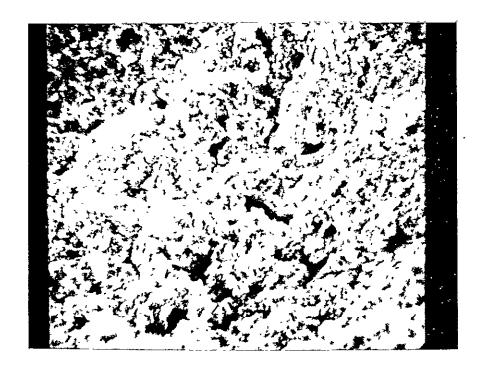


Silver Oxide Plate No. 120

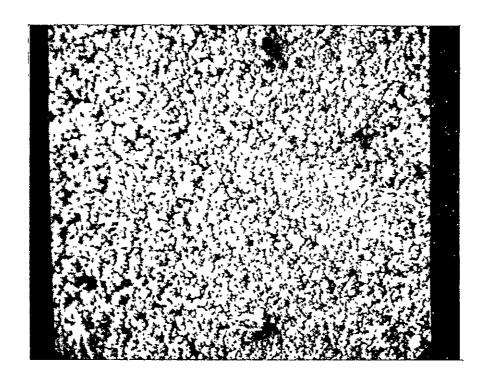
As Received

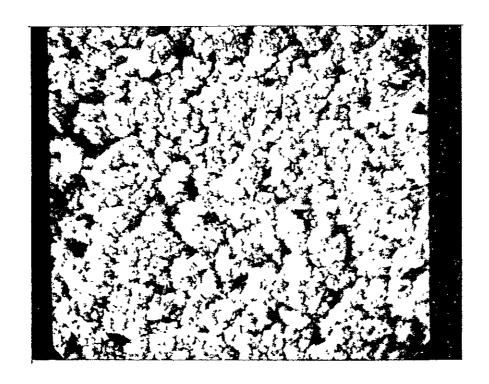
Transverse View





Silver Oxide Plate No. 120
Sterilized
Flat Surface

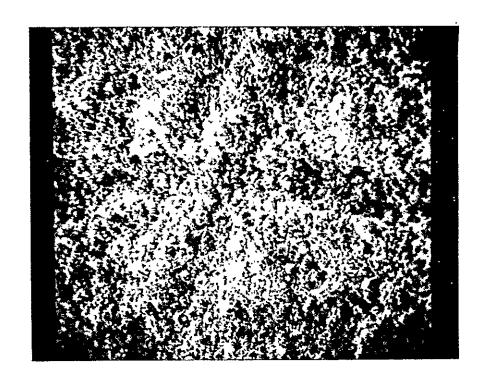


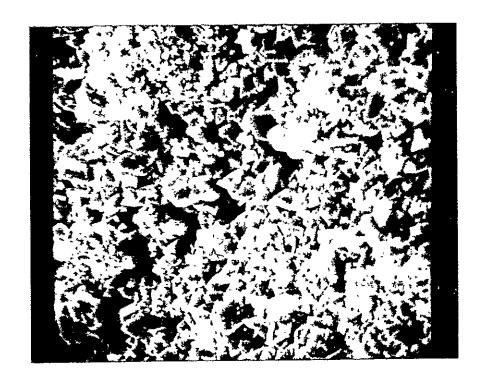


Silver Oxide Plate No. 171

As Received

Flat Surface

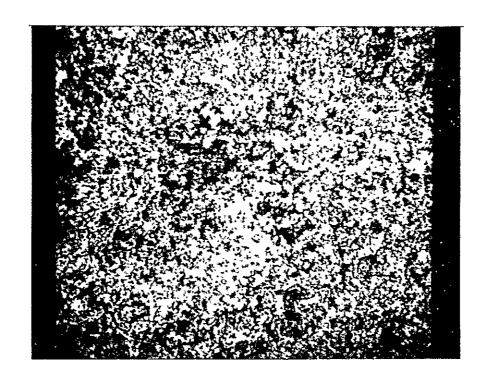


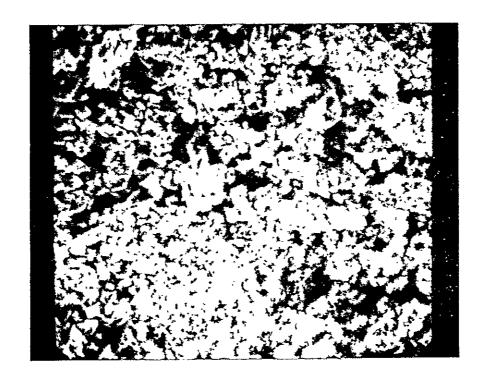


Silver Oxide Plate No. 171

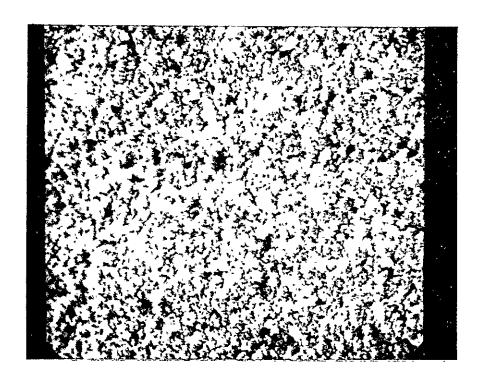
As Received

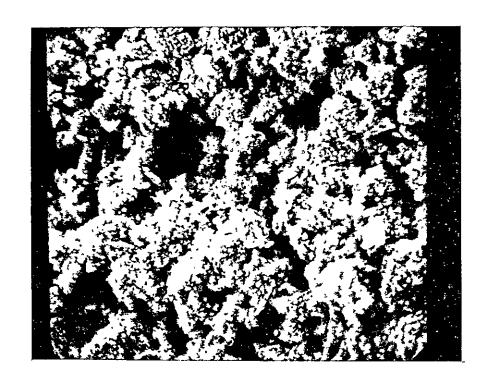
Transverse View



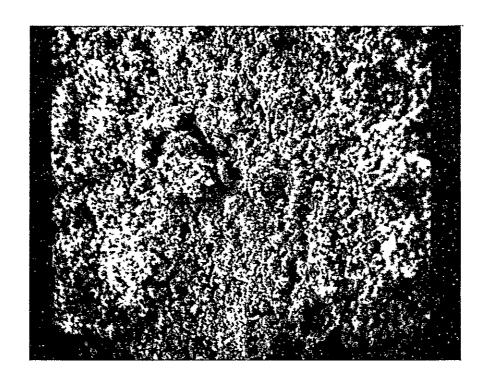


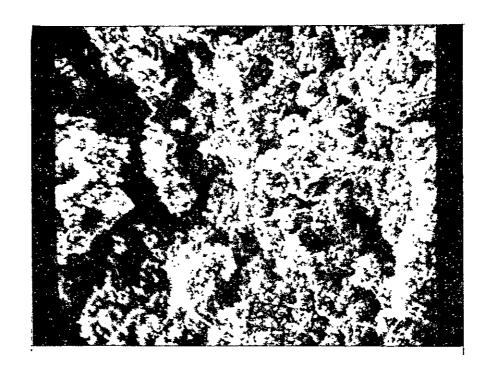
Silver Oxide Plate No. 171
Sterilized
Flat Surface





Silver Oxide Plate No. 206
As Received
Flat Surfáce

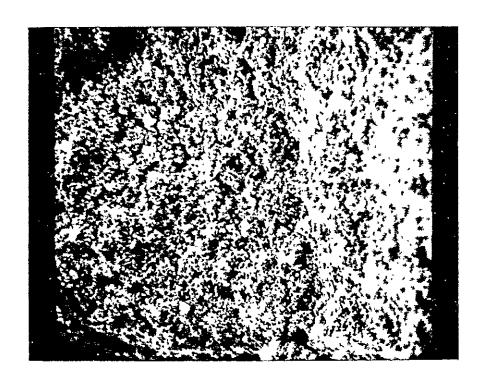


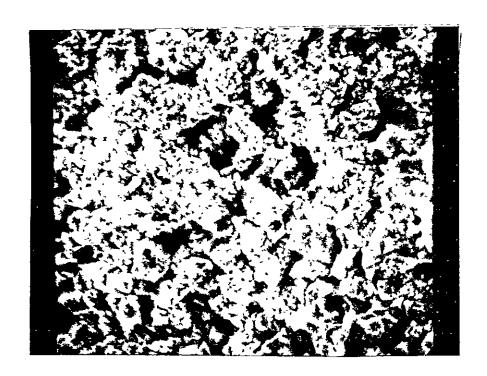


Silver Oxide Plate No. 206

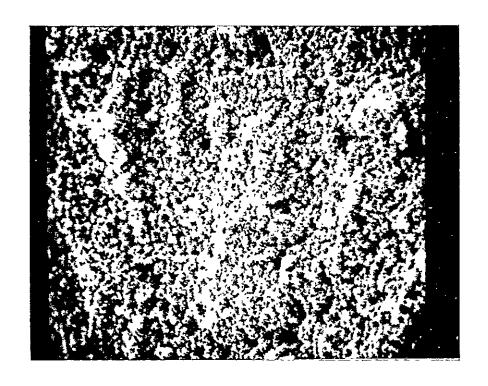
As Received

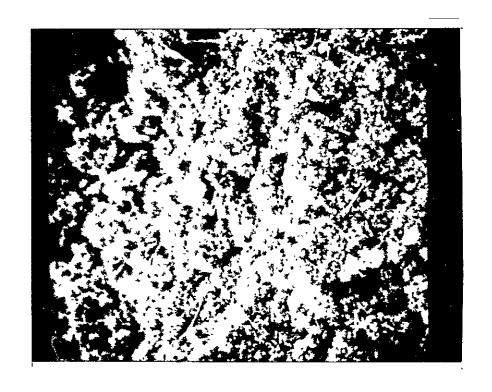
Transverse View





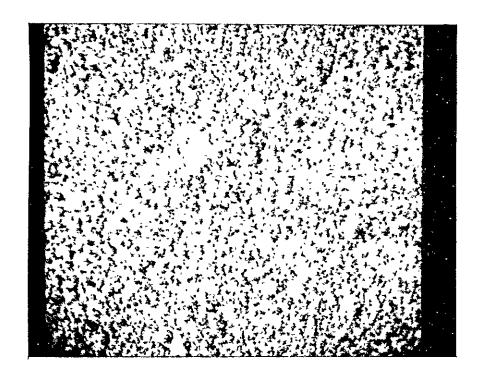
Silver Oxide Plate No. 206 Sterilized Flat Surface

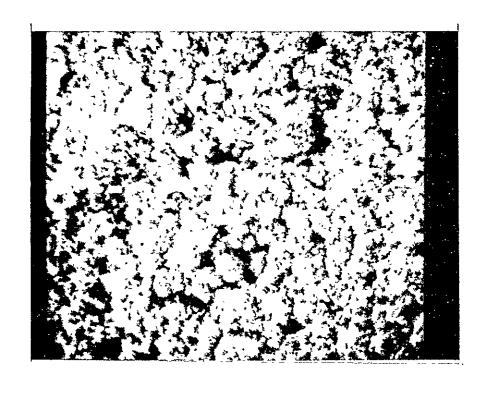




Silver Oxide Plate No. 252

As Received Flat Surface



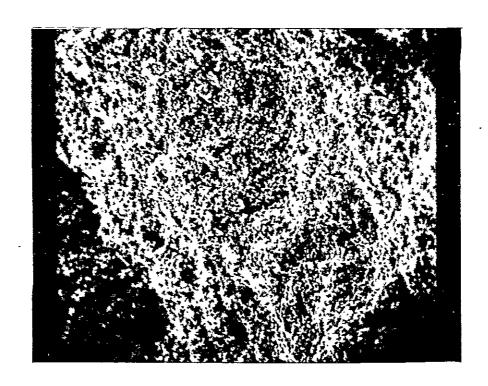


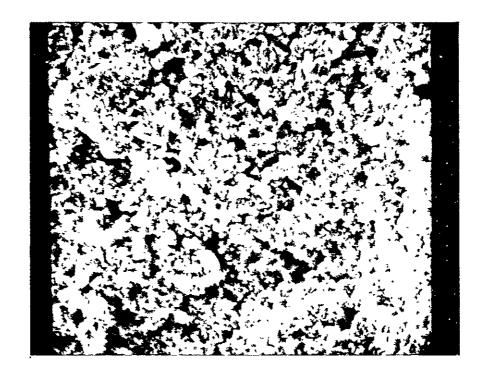
.

Silver Oxide Plate No. 252

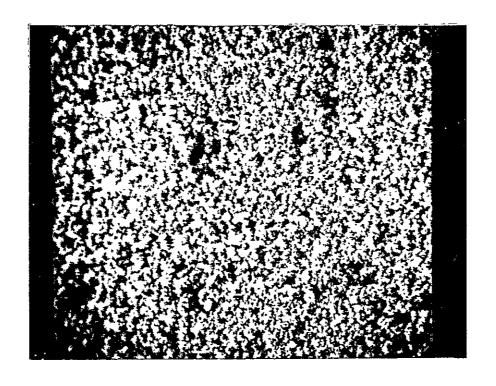
As Received

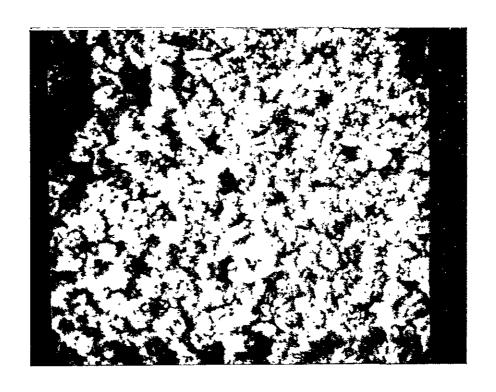
Transverse View



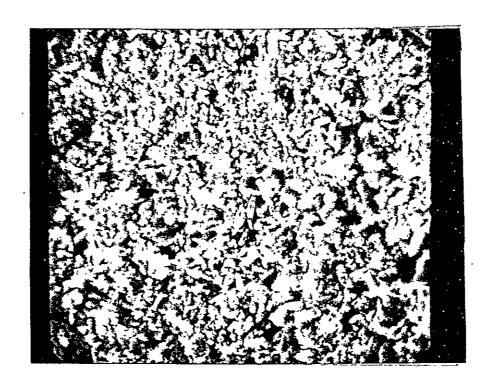


Silver Oxide Plate No. 252
Sterilized
Flat Surface



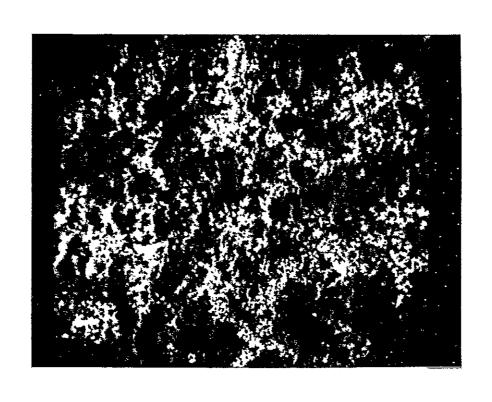


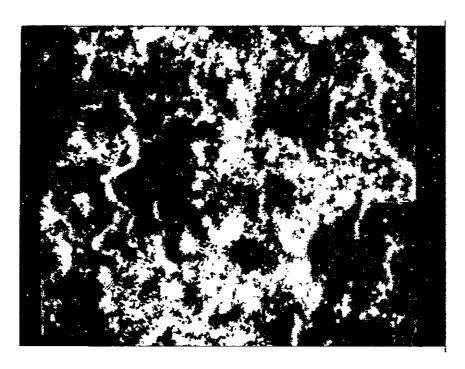
1000 Magnification
Silver Oxide Plate No. 252
Sterilized
Flat Surface



APPENDIX II MICROSCOPIC STUDY OF ZINC ELECTRODES

Zinc Plate No. 51
Sterilized
Flat Surface



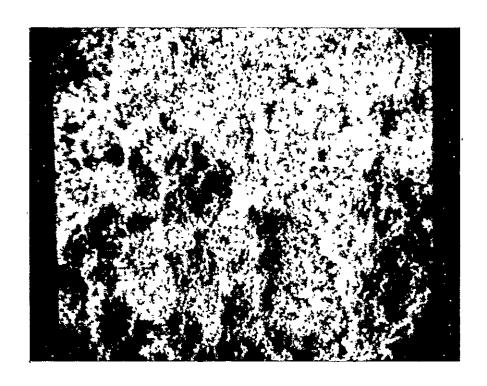


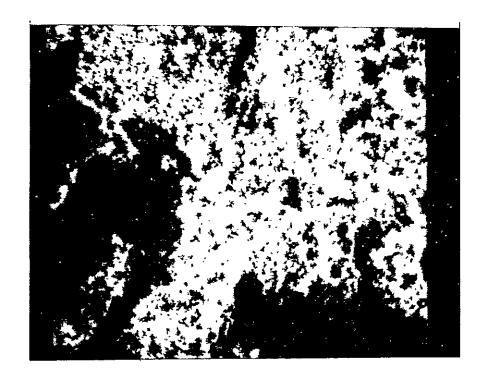
NOT REPRODUCIBLE

Zinc Plate No. 78

As Received

Flat Surface

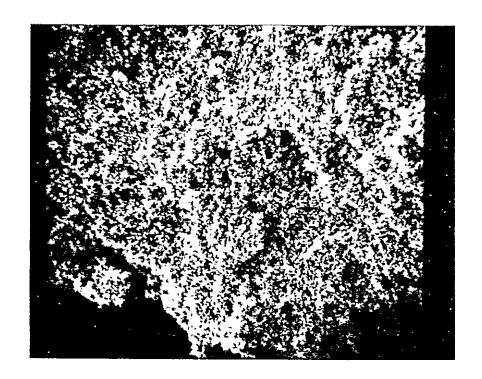


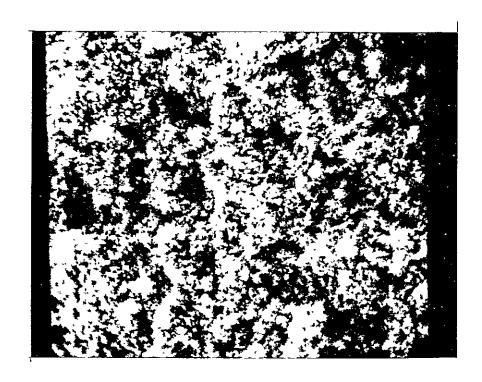


Zinc Plate No. 78

As Received

Transverse View

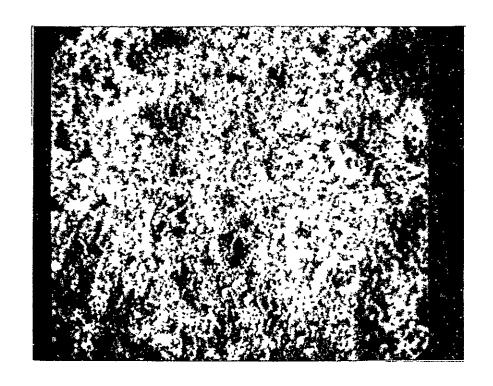


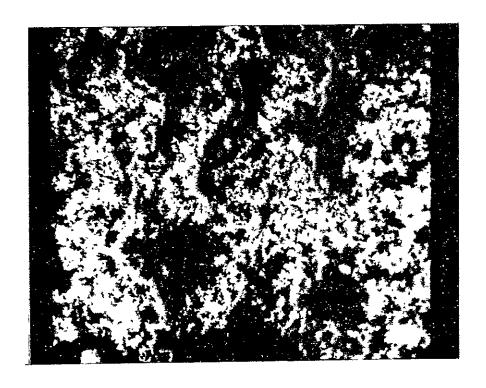


Zinc Plate No. 78
Sterilized
Flat Surface

1000 Magnification

-55-

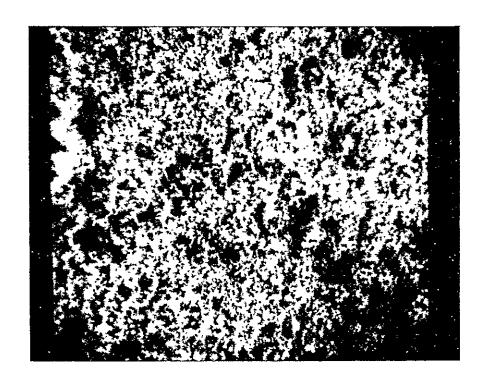


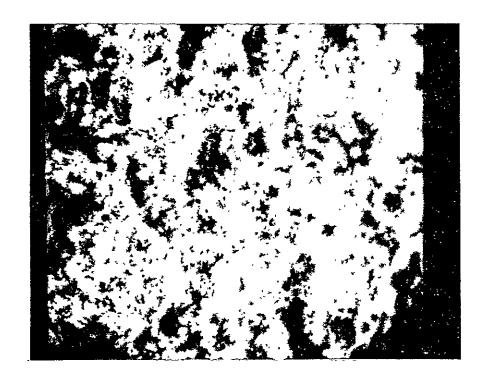


Zinc Plate No. 162

As Received

Flat Surface

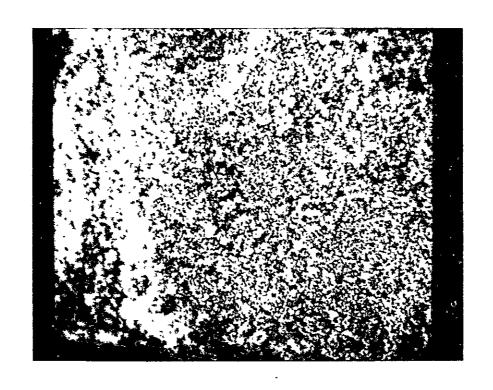


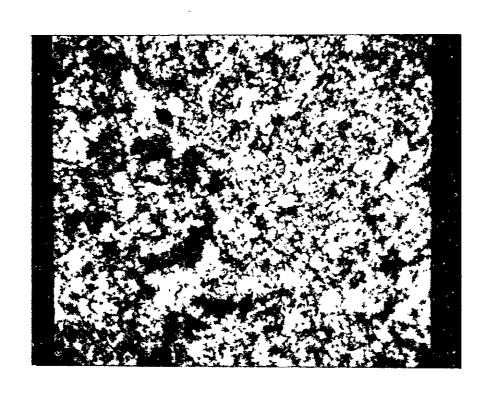


Zinc Plate No. 162

As Received

Transverse View





FNOT REPRODUCIBLE

Zinc Plate No. 162
Sterilized
Flat Surface

